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HAZARD CLASSIFICATION TESTING OF LIQUID PROPELLANTS

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19. ABSTRACT (CONTINUE ON REVERSE IF NECESSARY AND IDENTIFY BY BLOCK NUMBER)

The present TB 700-2 manual for establishing the hazard classification of energetic materials only addressed solid propellants and explosives in transportation and storage. The lack of a formal protocol for liquid propellants prompted the need for a program to provide the methodology, test procedures, test interpretations, and criteria for establishing a hazard classification. A methodology was submitted to the Safety Community for approval. The TB 700-2 manual tests were selected for evaluation as potential liquid propellant hazard classification candidates. Modifications had to be made to contain the liquid propellants for testing. This report addresses the small scale mandatory and non-mandatory interim classification tests, the modification required, the test results, and recommendation for establishing a hazard classification for a liquid propellant.

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INTRODUCTION

The Department of Defense (DOD) safety community has no formal protocol for the hazard classification of liquid propellants. In the past, they relied upon the utilization of the The Department of the Army Technical Bulletin 700-2 (TB 700-2), "Explosives Hazard Classification Procedures," for classifying energetic liquid materials. Unfortunately, this manual was designed for the hazard classification of solid propellants and explosives in transportation and storage. The manual explicitly states the exclusion of liquid propellants; thus, the hazard classification of liquid propellants has been assessed by tests designed for solid energetic materials.

The advent of liquid propellants as a potential replacement for solid propellants in the 155-mm gun created a need to provide a hazard classification for two hydroxyammonia nitrate (HAN)-based propellants, LP 1845 and LP 1846. To develop a viable test procedure for the establishment of a hazard classification for liquid propellants, an in-depth literature survey was made of available tests, procedures, interpretation of data and criteria used to classify solid propellants. An assessment was made of this information to determine if these hazard classification procedures could be applied to evaluate liquid propellants.

The following manuals were selected to develop test procedures and pass/fail critiera.

NATO AOP-7	Allied Ordnance Publication "Manual of Tests for Qualification of Explosive Materials for Military Use", August 1986
UN document	"Recommendations on the Transport of Dangerous Goods: Tests and Criteria", First Edition, United Nations, New York, 1986
TB 700-2	Department of Defense, Department of the Army, "Explosives Hazard Classification Procedures", Technical Bulletin 700-2, 1986

The NATO AOP-7 manual cites a characterization scheme for the classification of liquid propellants. The qualification tests and procedures are described, but the criteria for acceptance has been omitted. In the UN document that only applies to energetic materials in transportation, it provides for the test procedures and interpretation of the data, but assumes competency on the part of the testing authority to have discretion in interpreting the results, change the test procedures and eliminate or require additional tests. The major drawback of this document is a lack of designating criteria to establish a hazard classification.

The TB 700-2 was chosen as the role model for establishing the hazard classification of liquid propellants. This manual not only provides the tests and procedures, but cites the criteria for establishing a hazard classification. Because the HAN-based liquid propellants are sensitive to decomposition by transition metals and incompatible with many other materials, modifications to the test apparatus had to be made. Since the tests in the manual were designed for solid propellants, containment of the liquid propellant in compatible containers had to be incorporated into the tests where required.

This paper will discuss the methodology approved by the safety community, tests selected for evaluation, test results, and the critera generated for consideration as establishing the hazard classification of liquid propellants.

DISCUSSION

A methodology (shown in Figures 1-3) was developed and found acceptable by the safety community. The methodology accounts for the different modes of ignition that a material can experience in the environment. In the case of liquid propellants where they are transported from a storage reservoir to a combustion chamber and a pumping operation is required, compression ignition and critical diameter tests become essential. As demanded by the safety community in the TB 700-2, mandatory interim classification tests must be conducted before a hazard classification can be approved. These mandatory tests include the following:

1. Thermal
2. Card Gap
3. Impact
4. Detonation
5. Ignition and Unconfined Burning

Contingent upon the application and associated hazards, the following additional tests may be required:

1. Adiabatic Compression
2. Critical Diameter
3. Flash Point
4. Minimum Pressure for Vapor Phase Ignition
5. Electrostatics

Liquid propellants 1845 and 1846 were evaluated in each of the above tests in accordance with the appropriate test description.

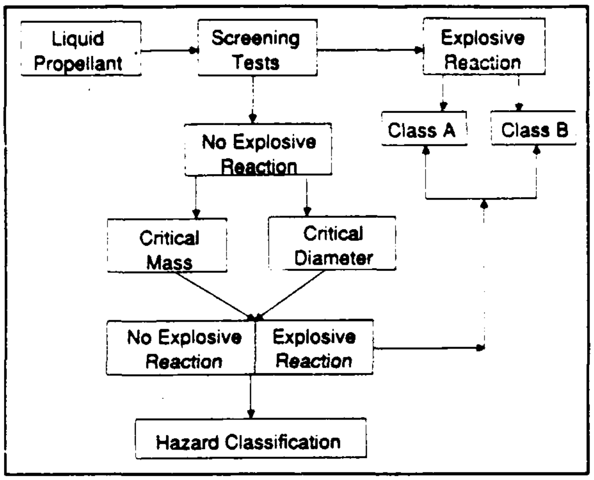

Figure 1. Hazard Classification Methodology

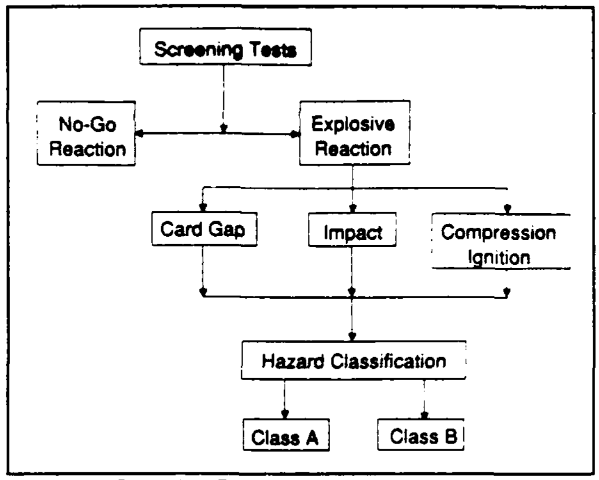

Figure 2. Explosive Reaction Methodology

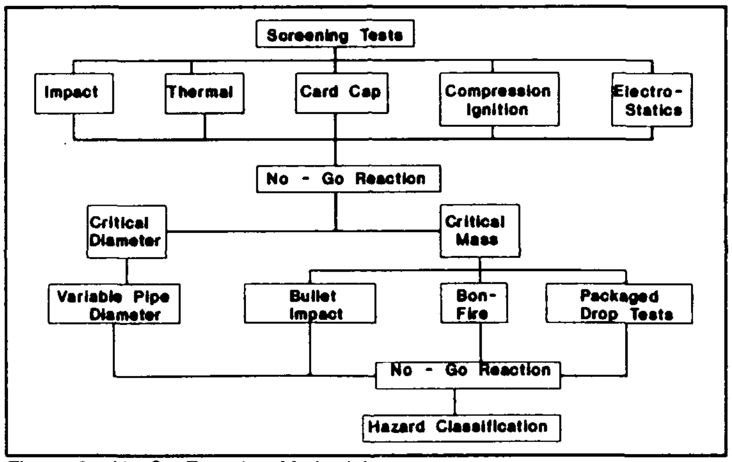

Figure 3. No-Go Reaction Methodology

TEST DESCRIPTIONS

THERMAL

Decontaminate a 2-inch diameter × 2-1/2-inch high × 0.5-mil thick polyethylene bottle filled with deionized water by placing in an oven at 71.1°C for 24 hours. Fill the decontaminated bottle with liquid propellant and place the liquid propellant-filled polyethylene bottle in a constant temperature, explosion-proof oven. Raise the temperature of the oven to 75°C and maintain this temperature for a period of 48 hours. Record any reactions that may occur over this period of liquid propellant exposure.

CARD GAP

The card gap test should be performed in accordance with the test procedures outlined in the TB 700-2. A typical card gap test set-up is shown in Figure 4. The test apparatus consists of a one piece 1.875-inch O.D. × 5.5-inch long mild steel tube. The ignition source consists of two pentolite pellets that weigh approximately 60 grams and a J-2 blasting cap. A 6-inch × 6-inch × 0.375-inch mild steel witness plate is used to determine if a detonation occurred. A detonation is indicated when a clean hole is cut into the witness plate. Cellulose acetate (or equivalent) cards 2 inches x 0.01 inch thick are used to attenuate a detonation. The greater the number of cards, the more sensitive the material. Four small pieces of plastic material cut into (0.0625 inch × 0.5 inch × 0.5 inch) pieces are used as shims to support the tube and maintain a 0.0625-inch air gap between the test sample and the witness plate.

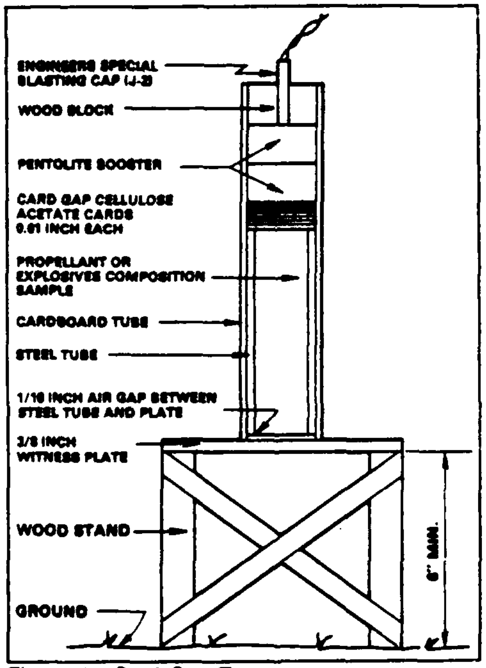

Figure 4. Card Gap Test

Liquid propellant, housed in a 1/2-mil thick polyethylene sleeve, is placed inside the mild steel tube. The sleeve is necessary to prevent any contamination of the liquid propellant. In the first test, the cellulose cards are omitted. Should no detonation occur in the first test, the test is repeated two more times. If a detonation occurs, then a series of tests (Figure 5) will be conducted using the cellulose acetate cards. The acetate cards are placed between the tube containing the liquid propellant and the pentolite booster. The first test is performed with eight cards. If a detonation occurs, the number of cards is doubled (add eight cards) for the next test. If a detonation occurs in this test, the number of cards is doubled again (add 16 cards). Continue doubling the number of cards until no detonation occurs. When no detonation occurs, then the number of cards is reduced by one-half from the preceding increment. As an example, if a detonation occurs at 32 cards but not at 64 cards, then the next test is run at 48 cards. If a detonation occurs at this reduced number of cards, the number of cards will be increased by one-half the preceding increment or 56 cards. The test procedure is continued until the point of 50% probability of a detonation is obtained. The measure of charge sensitivity is the length of the attenuation gap at which there is a 50% probability that a reaction has occurred.

IMPACT

The standard JANNAF drop weight tester was used to establish the impact sensitivity of liquid propellants. A typical test apparatus is shown in Figure 6. The liquid test sample is enclosed in a cavity formed by a steel cup, and elastomeric O-ring, and a steel diaphragm. A 4.4 pound (2 kilogram) weight is dropped onto a piston from a height of 48 inches. If a positive result occurs, the weight is dropped from a height one-half the original height. This adjustment of the drop height to one-half the distance is continued until no reaction occurs. A positive result has occurred when the steel diaphragm is punctured with an accompanied loud report, severe deformation of the diaphragm has occurred or evidence that the sample is consumed. Data are reported at the height which yields 50% probability of initiation.

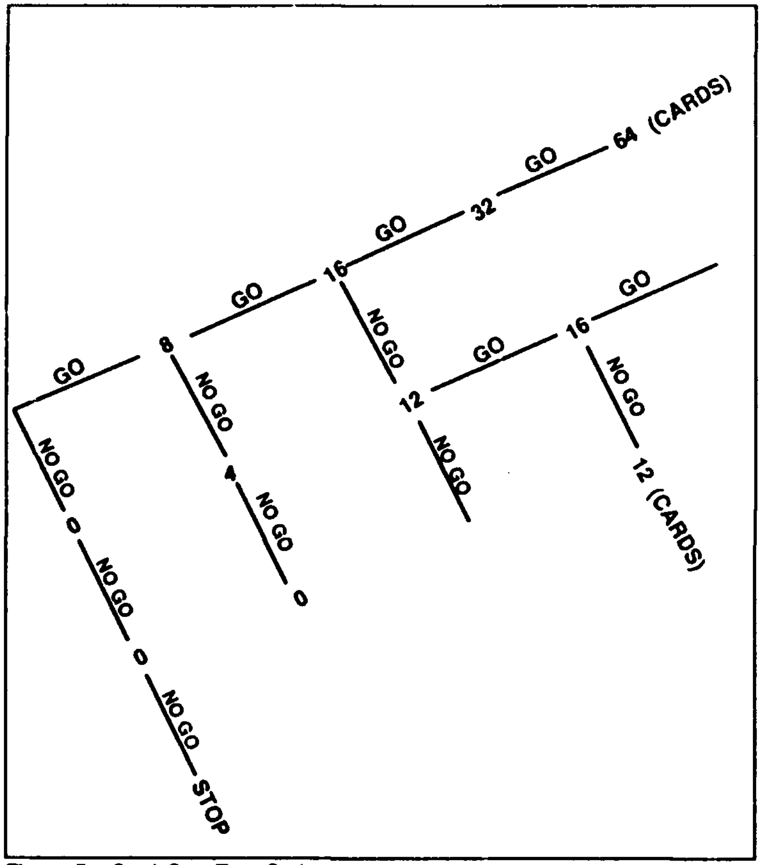

Figure 5. Card Gap Test Series

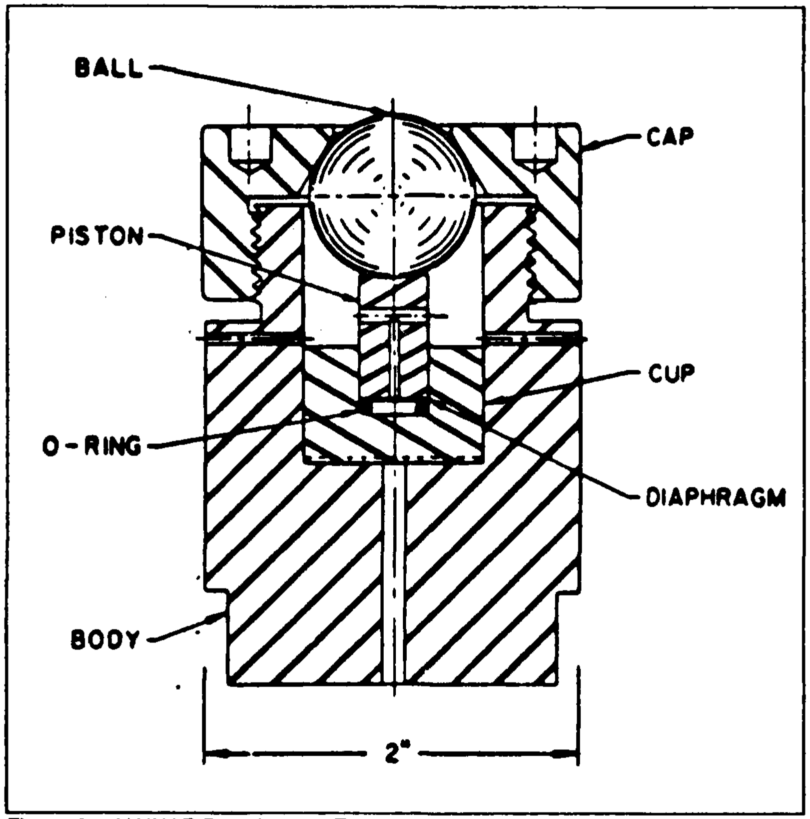

Figure 6. JANNAF Drop Weight Tester

DETONATION

A lead cylinder, 1-1/2 inch diameter × 4 inches high, is placed upon a 12-inch square × 1/2-inch thick SAE 1010 mild steel plate. Fill a decontaminate polyethylene bottle 2.5 inches high × 2 inches in diameter with a liquid propellant sample. A No. 8 blasting cap with the following requirements is placed perpendicular and in contact with the liquid surface:

1. A cap containing 0.4-0.45 grams of PETN base charge pressed into an aluminum shell having a bottom thickness not exceeding 0.03 inches

2. A specific gravity not less than 1.4 grams/cubic centimeter

3. Primed with standard weights of primer in accordance with the manufacturer's specifications

In the center of a wooden block, drill a hole and position the blasting cap. Ignite the blasting cap and examine the lead cylinder for any deformation. Any deformation of the lead block that is 1/8 inch or more will be considered evidence that a detonation has occurred. Conduct the test a minimun of five tests more or until a detonation occurs.

IGNITION AND UNCONFINED BURNING

Kerosene-soaked sawdust is placed in a 12-inch × 12-inch × 4-inch stainless steel container with a 1/16-inch wall thickness. The sawdust is evenly filled to a level of 1/4-inch thick. A 2-inch diameter decontaminated polyethylene bottle is filled with 2-1/2-inch height of liquid sample. The polyethylene filled test sample is placed in the center of the kerosene-soaked sawdust and ignited with an electric match-head ignitor. This test is repeated twice.

Four liquid propellant-filled decontaminated bottles are place in the center of a container filled with kerosene soaked sawdust. The bottles are placed in a row with each bottle in contact with the next bottle. The sawdust is ignited at one end with an electric match-head ignitor. The test is repeated two more times.

ADIABATIC COMPRESSION

The schematic of the U-tube compression ignition test set-up is illustrated in Figure 7. The following test parameters are required:

U-tube radius	1.0 inch
Sample volume	3.0 cubic centimeters
U-tube height	9.0 inches
Ullage space	6.0 inches
Tubing material	304 stainless steel
Tubing diameter	0.25 inch O.D. x 0.035 wall thickness
Pressure valve orifice	0.187 inches

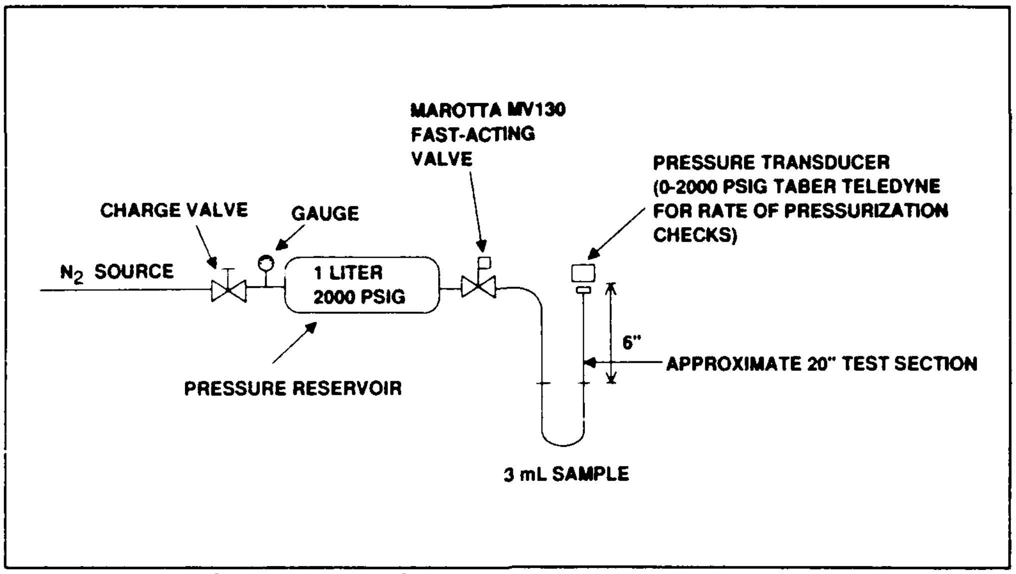

Figure 7. Adiabatic Compression Test Set-up

The U-tube is closed at one end with a cap. A 3-mL quantity of test fluid is placed in the curvature of the U-tube. The open end of the U-tube is connected to a discharge valve. A reservoir is pre-pressurized with nitrogen to 2000 psi. The test is conducted by suddenly pressurizing the U-tube. The pressure surge forces the liquid in the curvature to violently compress the ullage space containing the liquid vapors into the closed end. This rapid rate of pressurization is sufficient to provide adiabatic compression. Rupture of the U-tube is an indication that an explosion has occurred. The test is repeated with nitromethane as the control and the test results are compared.

CRITICAL DIAMETER

A schematic of the critical diameter test set-up is shown in Figure 8. Tests are conducted using different size diameter cylinders with L/D ratios of 2:1. The LP tests were conducted using 2-, 3-, and 4-inch diameter cylinders. Each cylinder is welded to a 316 stainless steel 1/4-inch thick witness plate. This assembly was passivated in a nitric acid bath to remove any potential contamination. An explosive C-4 charge is placed on top of the open end of the container, filled with liquid, and separated from the liquid by 0.5-mil thick polyethylene sheet. As the size of the container increased from 2 to 4 inches, the explosive charge was proportionally increased to ensure that the same energy per unit area was maintained. The 2-inch tests were conducted with 160 grams of C-4, the 3-inch tests used 360 grams, and the 4-inch tests were performed with 640 grams. These C-4 charge weights were chosen to produce a fixed energy input of 3.11×10^5 Joules/sq in. Velocity probes were inserted into the cylinders to determine the shock wave velocity as it travels through the liquid. The explosive charge is ignited and any reaction occurring is recorded.

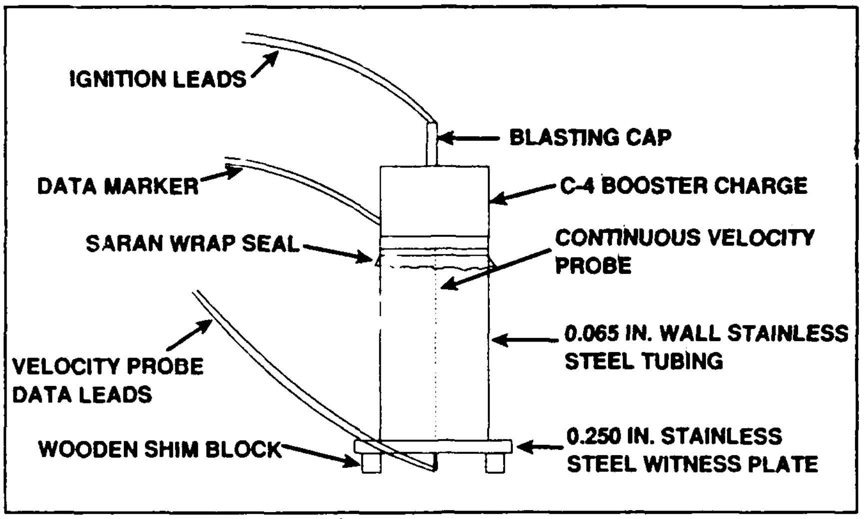

Figure 8. Critical Diameter Test Set-Up

FLASH POINT

The standard procedure for the ASTM-92-72 Cleveland open cup flash point test method was followed to evaluate the liquid propellants. The standard brass cup used in this procedure was replaced by a Pyrex® cup that reduced the standard volume from 70 mL to approximately 50 mL. The cup was filled with liquid propellant and placed in the test apparatus. The cup containing the fluid was heated to 25°C. A gas-fired flame was passed over the liquid. If no flash occurred, the temperature was raised in increments of 10° to a maximun of 75°C. The lowest temperature where the pilot flame caused the vapors above the liquid to ignite was taken as the flash point.

MINIMUM PRESSURE FOR VAPOR PHASE IGNITION

A schematic of the pressure test vessel for minimum pressure for vapor phase ignition is depicted in Figure 9. Water is used to calibrate the vessel. The vessel is evacuated and 2 mL of distilled water is injected into the vessel. The temperature is slowly raised at a rate of 5° per minute. At the 5° intervals, record the temperature and pressure. *n*-Propylnitrate is used as a standard for comparison. A fuse wire, 3-mL in length, is installed in the vessel such that 1 mL is unsupported. The vessel is evacuated and 2 mL of *n*-propylnitrate is injected into the vessel. The temperature of the liquid is raised to a temperature of 160°C (use caution). Record the temperature and the pressure at each temperature level recorded. Reduce the pressure to 2.2 atmospheres (29 psia). Ignite the vapor with the fuse wire and record the voltage, current and pressure. Repeat the test two more times.

The apparatus is cleaned with distilled water. A 3-mL sample of liquid propellant is injected into the vessel. The temperature is slowly raised, 5° per minute, until 100°C is reached; attempt ignition. Record voltage, current and pressure at this temperature. If no ignition occurs, discontinue test and restart test with procedure cited. Raise temperature 5° and attempt ignition. If no ignition occurs, repeat the test until ignition or a reaction occurs. At each temperature, record the voltage, current, and pressure.

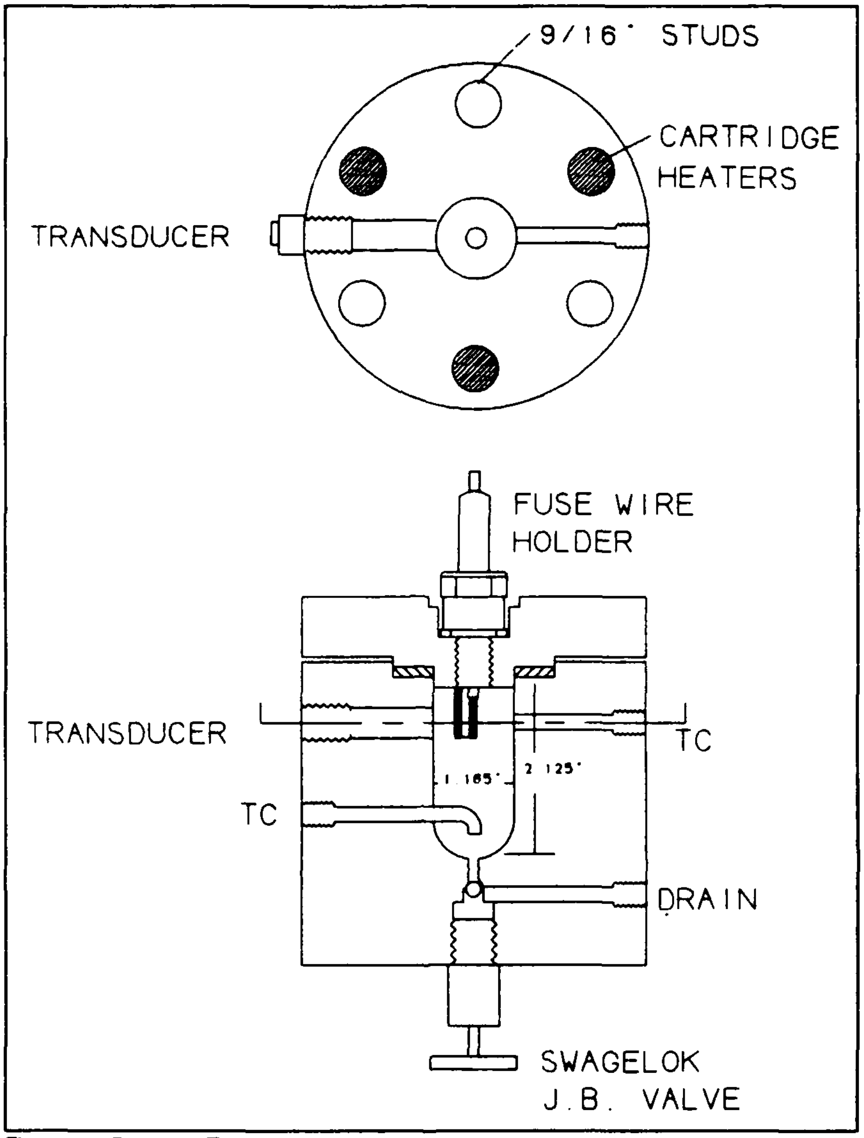

Figure 9. Pressure Test Vessel

ELECTROSTATICS

Electrostatic energy stored in a charged capacitor is discharged to the sample material to determine whether the electrostatic discharge will cause a sample to decompose, flash, burn, etc. The liquid sample is placed in a stainless steel 316 sample holder or equivalent material compatible with the liquid propellant that will permit the discharge to pass through the sample. The capacitor is to a 5000-volt potential. A discharge needle lowered above the liquid until a spark is drawn through the sample (20 mg). The standard test interval ranges from 0.0001 microfarads (μf) and 0.00125 J at 5 kV to 1 μf and 12.5 J at 5 kV. The test is initiated at the 1 μf and 12.5 J level. If a negative result occurs, testing is continued until 20 consecutive negatives are reported at that level. If the result is positive, such as a flash, spark, burn, odor or noise other than the instrument noise, a lower discharge level is selected until 20 or more negative results occur. The test voltage of 5 kV DC or less at ambient temperatures between 18.3°C and 32.2°C and a relative humidity not to exceed 40%.

RESULTS AND DISCUSION

The mandatory interim classification test results for thermal, card gap, impact, detonation and ignition and unconfined burning are discussed.

THERMAL

The results of the thermal test are found in Table 1. The Department of Transportation uses this test as a basis for "forbidden" materials for transportation. Had a detonation occurred in this test, the material could not be shipped. In both liquid propellants, LP 1845 and LP 1846, no reaction occurred after exposure to the test conditions.

TABLE 1. THERMAL STABILITY AND JANNAF THERMAL STABILITY TEST RESULTS

Test	Sample ID	Reaction	Temp of Major Major Exotherm Onset (°C)
Thermal Stability[a]	LP 1845	None	
	LP 1846	None	
JANNAF Thermal Stability[b]	LP 1845		120
	LP 1846		122

a. 48 hours at 75°C in vented oven.
b. Heat at constant temperature rate of 10°C/min

CARD GAP

The card gap test results are found in Table 2. In testing LP 1845 and LP 1846, zero cards were used which is indicative of the insensitivity of these materials. Under the criteria for a solid propellant, 70 cards or below would classify this material as a Class B. Had the result been in excess of 70 cards, the material would have been classified as a Class A. The diameter of the card gap test tube is below the critical diameter of these materials (see critical diameter test results). It is felt that at this diameter, the material will not propagate a shockwave to detonation. The length of cylinder is also only 5.5 inches which may not provide enough residence time for a reaction and it has been suggested to increase this length to 16.5 inches.

TABLE 2. CARD GAP DATA

Sample ID	No. of Cards	Visual Observation
LP 1845	0	Witness plate deformed No holes in plate
LP 1846	0	Witness plate deformed No holes in plate

IMPACT

The impact test data can be found in Table 3. As one might expect, the difference in drop height between LP 1845 (28 inches at 0%) and LP 1846 (29 inches at 0%) is only 1 inch. This can be attributed to 3% more water in LP 1846. Nitromethane, classified by the Department of Transportation as a flammable liquid, has a drop height of 20 inches. As one can see, the LP materials are less sensitive than nitromethane.

TABLE 3. IMPACT TEST DATA

Sample ID	Drop Height (in.) 0%	50%	100%
LP 1845	28	30	31
LP 1846	29	30.5	33

DETONATION

Detonation test data are shown in Table 4. In LP 1845 and LP 1846, no detonations occurred. This test is considered by the Department of Defense (DOD) as one of their critical mandatory test requirements. A detonation here would immediately characterize the material as mass detonable or Class A and a 1.1 designation.

TABLE 4. DETONATION TEST RESULTS

Sample ID	Detonation Reaction
LP 1845	None
LP 1846	None

IGNITION AND UNCONFINED BURNING

The test results from the ignition and unconfined burning are depicted in Table 5. LP 1845 and LP 1846 indicated no reaction when exposed to burning sawdust soaked with kerosene. This test simulates a fire occurrence in storage of a liquid propellant and whether a thermal heat transfer can initiate a detonation.

TABLE 5. UNCONFINED BURNING TEST RESULTS

Sample ID	Detonation Reaction
LP 1845	None
LP 1846	None

ADDITIONAL TESTS

The next series of test results are not mandatory, but may be required by the safety community. This requirement is contingent upon the particular application that the liquid propellant must be exposed.

Adiabatic Compression

Adiabatic compression test results are shown in Table 6. No detonation occurred when a pressurization rate of 260,000 psi/sec was applied to LP 1845 and LP 1846. It is critical that the same configuration, described in the test description, be used for this test; otherwise, erroneous data will be obtained. It has been found that a slight change, such as replacing the end cap with a pressure transducer cap, will have a pronounced effect upon the test result. The standard end cap which has a conical interior produced a detonation with nitromethane; whereas, when replaced with a flat interior transducer cap, no detonation occurred. Both tests were conducted at the same pressurization rate. It is theorized that this is a heat transfer problem associated with the conical interior cap. A bubble trapped in the conical space affords a hot spot for a reaction to occur.

TABLE 6. ADIABATIC COMPRESSION TEST RESULTS

Sample ID	Reaction
Control (water)	None
LP 1845	None
LP 1846	None
Nitromethane	Detonation

Critical Diameter

The LP 1845 and LP 1846 test data for the critical diameter tests are found in Table 7. Detonation probes indicated that no detonation occurred with LP 1845 in a 3-inch diameter cylinder, but a detonation wave was detected in the 4-inch diameter cylinder. LP 1846 was less sensitive since no detonation was detected in the 4-inch diameter cylinder; however, it produced a detonation wave in the 5-inch diameter cylinder. This differential in the degree of sensitivity for LP 1846 can be attributed to 3% more water in the basic stoichiometric formulation. The 3:1 charge height to diameter was not followed from the standard test because it was felt that any deviation in the planer wave across the diameter from this test charge configuration would be negligible.

TABLE 7. CRITICAL DIAMETER TEST DATA

Sample ID	Baffles*	Detonation Reaction
LP 1845	No	Apparent reaction at 4 in.
LP 1846	No	Apparent reaction at 5 in.
LP 1845	Yes	Apparent reaction at 4 in.
LP 1846	Yes	Apparent reaction at 5 in.

* Whiffle ball-type polyethylene spheres occupying approximately 12% of the canister volume

Flash Point

The flash point data for LP 1845 and LP 1846 are shown in Table 8. Since the basic stoichiometric formulation of each formulation contains 16.8% and 20.0% water in LP 1845 and LP 1846, respectively, the vapor above each compound is essentially water. Therefore, one would expect that there would be no flash point, and this was the case for both materials.

TABLE 8. FLASH POINT TEST RESULTS 75°C (Propane Flame)

Sample ID	Reaction
LP 1845	None
LP 1846	None

Minimum Pressure for Vapor Phase Ignition

Minimum pressure for vapor phase ignition test results for LP 1845 and LP 1846 are given in Table 9. Here again in this test, the vapor above the liquid phase is water. In the test, the material began to decompose when the temperature reached 120°; thus, the is no minimun pressure for vapor phase ignition. The material simply does not ignite under these conditions.

TABLE 9. MINIMUM PRESSURE FOR VAPOR PHASE IGNITION TEST RESULTS

Sample ID	Reaction
Water (control)	None
LP 1845	None (material decomposed)
LP 1846	None (material decomposed)

Electrostatics

The electrostatic LP 1845 and LP 1846 test results are shown in Table 10. Both materials were unreactive to electrostatic ignition. This was anticipated since the component oxidizer, hydroxyammonium nitrate, and the fuel, triethanolammonium nitrate, are nitrated salts that are completely ionized in the water portion of the formulation. Thus, the electostatic charge build-up is readily dissipated and cannot materialize into a hazardous discharge.

TABLE 10. ELECTROSTATICS TEST RESULTS (1 μf and 12.5 Joules at 5 kV)

Sample ID	Reaction
LP 1845	None
LP 1846	None

SUMMARY

The completion of these small scale classification tests permits an application to the safety communuity for an interim classification for LP 1845 and LP 1846 in storage and transportation. The present classification for these liquid propellants is a 1.3 Class B explosive. The insensitivity of this material may warrant a Class C designation. The large-scale packaged end item tests will finalize the classification of LP 1845 and LP 1846. Ultimately, this work effort will result in a liquid propellant hazard classification manual that will be incorporated into the DOD TB 700-2.

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